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Indian Standard

PREPARATION OF COPPER AND COPPER-BASE ALLOYS FOR ELECTROPLATING — RECOMMENDED PRACTICE

(First Revision)

UDC 669·3-4 : 621·357·5

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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Metallic and Non-Metallic Finishes Sectional Committee, MTD 20

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards on 20 October 1989, after the draft finalized by the Metallic and Non-Metallic Finishes Sectional Committee had been approved by the Metallurgical Engineering Division Council.

This standard was first published in 1980. In view of the experience gained and the latest developments in the field of electroplating, it was felt necessary to revise this standard. In this revision, the alkaline electroclean cycle in preparatory cycles has been modified to cathodic alkaline electrocleaning apart from other modifications in the preparatory cycles.

This recommended practice is intended as an aid to platers in setting up a suitable cleaning and conditioning cycle preparatory to electroplating on copper and copper base alloys. It is noted as a standardize procedure and should be used merely as a guide. The sequence of the individual cycles and the concentrations of the various solutions are flexible and may be varied to fit the conditions of the individual plating department. The purpose of this cycles is to prepare the work to take a sound, adherent electroplated coating.

In the preparation of this standard, assistance has been derived from ASTM Designation: B 281-1982 'Standard practice for preparation of Cu and Cu base alloys for electroplating and conversion coatings' published by the American Society for Testing and Materials.

Indian Standard

PREPARATION OF COPPER AND COPPER-BASE ALLOYS FOR ELECTROPLATING — RECOMMENDED PRACTICE

(*First Revision*)

1 SCOPE

1.1 This standard describes procedure for preparation of copper and copper alloys surfaces for electroplating. It, however, does not cover powdered metal parts of copper alloys. Only alloys containing at least 50 percent copper by mass, considered within the scope of this practice.

2 PREPARATION OF SURFACE

2.1 Removal of Oxides

The removal of oxides from cast, annealed, or heat-treated copper and copper base alloys may be accomplished by abrasive methods, such as tumbling, burnishing, emery set up wheel polishing blasting, buffing, and greaseless compound buffing, and by chemical methods, such as pickling, bright dipping 'fire off' or scale dipping, and cyanide dipping. The choice of methods is dependent on the resultant surface finish required and on the amount and type of oxide to be removed.

2.2 Castings and Forgings

Castings and forgings requiring abrasive methods to produce a desired surface finish do not necessarily need pickling or bright dipping. However, if pickled or bright dipped, castings and other porous parts should be carefully treated to avoid strain spots; these products should be very thoroughly rinsed between operations in hot and cold water. Water repelling organic dips are also useful in removing residual chemicals which might cause staining. Castings or forgings processsed in solutions containing wetting agents, which are in many proprietary products or may be added by the individual, usually require great care in rinsing.

NOTE — 'Spoting out' may occur after castings have been plated. This is usually due to porosity in the castings and inadequate rinsing between the various steps of the cycles. Acid or cyanide solutions may be retained in the pores of the metal. To help in the removal of cyanides or acids from the pores, several alternate hot and cold rinses are used, allowing the work to come to the temperature of the water before each removal. The most satisfactory method of eliminating those troublesome defects is to eliminate the cause that is porous basis metals.

2.3 Stampings and Drawn Products

Stampings and drawn work follow the same rule as castings and forgings, except that for economic reasons it may be advisable to pickle before abrasive finishing if heavy oxides are present due to annealing. For example, excessive amounts of compounds or wheels may be consumed in removing oxides in the buffing operation before the actual work of smoothing the surface begins.

2.4 Screw Machine Products

Screw machine products may be plated as they come from the machines, or abrasive finishing methods may be applied.

2.5 Cold-Headed and Multiple-Plunger Press Products

Cold-headed products and multiple-plunger press machine products often require relief annealing to avoid subsequent season cracking. This annealing leaves some oxides which may be removed by pickling.

3 OUTLINE OF PREPARATORY CYCLES

3.1 Buffed Work

Following is a typical cycle to be used for plating copper and copper-base alloys which have been buffed and are to be plated in an acid solution such as bright nickel or acid copper:

a) The buffed component may first be soaked in kerosene oil to loosen the polishing compound. Then vapour or solvent degrease (if heavy soil is present) or alkaline or emulsion soak clean;

b) Rinse;

c) Alkaline electroclean (Cathodic);

d) Rinse (*see* Note);

e) Dip in acid;

f) Rinse; and

g) Plate in acid solution

NOTE — A cyanide dip and rinse may be used to advantage between steps (d) and (e).

3.2 Following is a typical cycle for buffed work to be plated in an alkaline solution:

a) The buffed component may first be soaked in kerosene oil to loosen the polishing compound. Then vapour or solvent degrease (if heavy soil is present) or alkaline or emulsion soak clean;

- b) Rinse;
- c) Alkaline electroclean (Cathodic);
- d) Rinse (*see* Note);
- e) Dip in cyanide solution;
- f) Rinse, or copper strike and rinse; and
- g) Plate in alkaline solution.

NOTE — An acid dip and rinse may be used to advantage between steps (d) and (e).

3.3 Following is a cycle for buffed and soldered or brazed parts or screw machine work of leaded alloy to be plated in acid solution such as bright nickel or acid copper:

- a) Vapour or solvent degrease (if heavy soil is present) or alkaline or emulsion soak clean),
- b) Rinse,
- c) Alkaline electroclean (cathodic),
- d) Rinse (*see* Note),
- e) Dip in cyanide solution (*see* Note 1),
- f) Rinse (*see* Note 1),
- g) Strike in copper (cyanide) (*see* Notes 1 and 2) or low *p*H nickel electrolyte (*see* Note 1),
- h) Rinse,
- j) Dip in acid,
- k) Rinse, and
- m) Plate in acid solution.

NOTES

1 A dip in fluoboric or dilute nitric acid followed by a rinse, may be used to advantage between steps (d) and (g) if a copper strike used, or in place of steps (e) and (f) if a nickel strike is used.

2 A cyanide solution should not be allowed to accumulate lead, which may cause lead immersion plate with subsequent blistering.

3.4 Bright Dipped or Pickled Work

Following is a typical cycle for work to be plated in acid solution:

- a) Degrease (if this stage is necessary, usually an alkaline spray or soak will suffice),
- b) Rinse,
- c) Alkaline electroclean (cathodic),
- d) Rinse (*see* Note),
- e) Dip in acid (*see* Note),
- f) Rinse (*see* Note), and
- g) Plate in acid solution.

NOTE — A bright dip may be added between steps (d) and (e) and should be followed by a thorough agitated and running rinse before step (e). Usually however, the bright dip and rinses replace steps (e) and (f). Electropolishing of copper-base alloys may also be used in place of bright dipping.

3.5 Following is a typical cycle for work to be plated in an alkaline solution:

- a) Degrease (if heavy soil is present) or alkaline or emulsion clean,
- b) Rinse,
- c) Alkaline electroclean (cathodic *see* Note 1),
- d) Rinse (*see* Note 2),
- e) Dip in cyanide (*see* Note 2),
- f) Rinse, and
- g) Plate in alkaline.

NOTES

1 *Electrocleaning* — To produce a chemically clean surface required for electroplating of subsequent coatings. an alkaline eletcrolytic cleaner may be used, making component as the anode or cathode — Anodic cleaning, particularly of brass, may cause slight tarnishing or etching if applied for a prolonged time (more than a few seconds) or at too high or too low in operating temperature. A contaminated cathodic cleaner may form a smut film on the work. The working voltage at the source may be 6 to 8 V. The general practice is in favour of cathodic cleaning.

2 A bright dip may be added between steps (d) and (e) and should be followed by two agitated and running rinses before step (e). Electropolishing of copper-base alloys may also be used in place of a bright dip.

3.6 For details of typical cleaning and pickling solutions, Annex A may be referred.

4 WORK TO BE BARREL PLATED

4.1 Work to be barrel plated may be alkali-cleaned without current by tumbling. However, if electrocleaning facilities are available, they should be used.

4.2 Following is a typical cycle for barrel work to be plated in an acid solution (*see also* Note below):

- a) Vapour of solvent degree in bulk if heavy soil is present or alkaline soak clean,
- b) Rinse in bulk,
- c) Clean in barrel (electrolytically or by immersion and tumbling) (*see* Note),
- d) Rinse (*see* Note),
- e) Acid dip or bright dip (*see* Note),
- f) Rinse (*see* Note), and
- g) Barrel plate in acid solution.

NOTE — Steps (c) to (f) inclusive may be done in bulk in suitable containers such as baskets if proper precautions are used to ensure thorough cleaning and rinsing at each step. There is available equipment so designed that the work is treated in barrels during all operations from cleaning through plating and in many cases this may be done in the plating cylinder. Step (e) may be followed by ball burnishing in which case step (c) to (f) inclusive, should be repeated before plating. A cyanide dip and rinse may be used to advantage between steps (d) and (e).

4.3 Following is a typical cycle to be used for barrel work to be plated in an alkaline solution:

- a) Vapour or solvent degrease in bulk if heavy soil is present,

b) Rinse in bulk,

c) Clean in barrel (electrolytically or by immersion and tumbling; a cyanide dip may be used),

d) Rinse [(*see* Note under **4.2** (g) and **4.3** (g)],

e) Dip in cyanide [*see* Note under **4.2** (g) and **4.3** (g)].

f) Rinse, and

g) Plate in alkaline solution.

NOTE — An acid dip, followed by a rinse, may be used to advantage between step (d) and (e).

4.4 For details of typical cleaning and pickling solutions. Annex A may be referred.

5 DETAILS OF PREPARATORY OPERATIONS

5.1 Cleaning

The first step in the preparatory cycle is cleaning. The soil to be removed determines the type and complexity of the method or methods to be used. One stage or a series of a stages may be employed to do the cleaning. Following is a listing with a brief description of the various stages available.

5.1.1 *Oil and Grease Removal*

Degreasing is used to remove the gross accumulation of dirt, grease, and oil and when properly performed removes most of the soil. The removal of heavy impacted buffing compound may require use of a soak in hot or cold solvent or hot solvent emulsion cleaner prior to subsequent cleaning. Oil and grease removal is generally accomplished by use of vapour degreasers (trichloroethylene or perchloroethylene); or immersion in soak tank (work preferably agitated) containing emulsion cleaners, di-phase cleaners or alkaline cleaners; and spray washing machines employing emulsion or alkali cleaners.

5.1.2 *Electrolytic Alkaline Cleaning*

To produce the chemically clean surface required for electroplating, an electrolytic cleaner is used. Electrolytic cleaners are used with the work as the anode only or with the work as the cathode followed by the work as the anode. Anodic cleaning, particularly of brass, may cause slight tarnishing or etching is applied for a prolonged time (more than a few seconds) or at too high or too low a temperature. A contaminated cathodic cleaner may form a smut on the work. The voltage at the source is often 6 to 8 V. Separate solutions should be used for anodic and cathodic cleaning. Electrolytic cleaner composition is an extremely complex subject. The formula below is one of many (the chemicals are technical grade):

	Mass, percent
Sodium carbonate (Na_2CO_3)	40 to 50
Trisodium phosphate (Na_3PO_4, $12H_2O$)	25 to 40
Sodium hydroxide (NaOH)	10 to 25
Surface active agent (to provide a foam blanket)	1 (approximately)

Total of individual ingredients adjusted to give 100 percent

This mixture is used in a solution concentration of 30 to 45 g/l

Temperature	60 to 70°C
Current density	1 to 3 A/dm^2
Time	1 to 3 minute cathodic and 5 to 10 seconds anodic

NOTE — Many prefer to use proprietary cleaners. These cleaners are used in accordance with the supplier's recommendations.

5.1.3 *Tumble Cleaning*

A typical tumble cleaner is listed below. Proprietary cleaners are available and are generally considered preferable:

	Mass percent
Mild soap chips or powder	12·5
Trisodium phosphate (Na_3PO_4, $12H_2O$)	12·5
Sodium carbonate (soda ash)	75

This mixture is used in a solution of concentration from 30 to 45 g/l. Warm mixture is preferable.

When a part is properly cleaned, it will show a continuous liquid film upon removal from rinse and acid dipping cycles.

6 TARNISH AND STAIN REMOVAL, NEUTRALIZATION

6.1 Acid Dipping

After the work has been thoroughly cleaned and rinsed, it should be acidified to neutralize any residual alkali before it enters an acid plating bath. Thus, prior to nickel, acid copper, chromium, fluoborate and similar plating solutions, an acid dip is used. The most common dips used are 5 to 10 or 10 to 20 volume percent of hydrochloric acid. Fluoboric acid, 5 to 10 volume percent of 42 to 45 percent strength may be used prior to fluoborate plating solutions. These dips are maintained at room temperature. The first two dips are usually held in containers that have been lined with rubber, plastic sheat or brick, or in crocks. The fluoboric acid should be contained in a rubber or plastic lines tank. A lead lined tank is satisfactory for the sulphuric acid. Leaded copper alloys, because they form insoluble salts in sulphuric or hydrochloric acid, should be pickled in fluoboric acid or nitric acid.

6.2 Cyanide Dipping

After the work has thoroughly cleaned and rinsed it is usually immersed in a sodium cyanide dip prior to plating in a alkaline solution. This dip will remove slight tarnish caused by cleaners. A concentration of 15 to 40 g/l may be used at room temperature.

6.3 Bright Dipping

Bright dipping is usually performed prior to the preparatory cycle but is sometimes included as a means of reactivation of the surface. However, with some jewellery plating, for example, it is an integral part of the preparation. Bright dipping is intended primarily to improve surface lustre of the work. It may also be used for removal of light oxides on forgings or castings. Maximum surface lustre (not mirror brightness) may be obtained by immersing the work with constant agitation for 5 to 10 seconds in one or several solutions, one of which follows:

60 to 75 volume percent sulphuric acid,

20 to 35 volume percent nitric acid,

5 to 10 volume percent water, and

1 g/l of concentrated hydrochloric acid.

The bright dipped parts should be rinsed thoroughly by immersion and constant agitation in two successive cold water rinse tanks. Small irregularly shaped parts, in which acid may become entrapped, should be immersed in a warm mild alkaline solution to neutralize the acid before immersion in cyanide. In excess of hydrochloric acid should be avoided; otherwise, a dull finish will result. Sometimes a small amount of lump black is added to improve the brilliance of the work. A stoneware crock is used as a container for the acid bright dip solution. It is important that the bright dip solution to kept cool; otherwise, the work will etch instead of becoming bright. Therefore, it is desirable to place the container setting in a running cold-water rinse tank. This also serves as safety feature, in case the crock should break. It is necessary to have adequate ventilation for these dips. If the solution is inhibited with 15 to 20 g/l of copper sulphate, a stainless steel container may be satisfactory.

7 STRIKING

7.1 Copper Strike

In order to eliminate peeling, many operators copper strike before silver or nickel plating leaded copper alloys and work which has been soft soldered. A low *p*H nickel strike may often be substituted for or used in addition to the copper strike, especially before silver plating. A standard cyanide copper strike may be employed. All solder and the basis metal should be completely covered with copper. This requires up to 1 minute at 3 to 6 V.

7.2 Silver Strike

As in plating any basis metal, it is necessary to apply a silver strike to the work before it enters a silver plating solution. The power source should be on before immersing the work in either silver strike or silver plating solution.

ANNEX A

(*Clauses* 3.6 *and* 4.4)

CLEANING AND PICKLING SOLUTIONS

A-1 FORMULAE FOR CLEANING AND PICKLING SOLUTIONS

A-1.1 Following are formulae which may be useful in preparing, cleaning and pickling solutions for copper and copper-base alloys for abrasive finishing in preparatory cycles outlined in **3** and **4** of this recommended practice.

A-1.2 Sulphuric Acid Pickle

For copper and copper-base alloys (except beryllium copper) to remove oxides, 10 to 40 volume percent sulphuric acid may be used. This solution is contained in a lead lined tank or tub. This solution may be used from room temperature to 80°C. Time required is approximately 10 minutes.

A-1.2.1 For beryllium copper to remove oxides, 60 to 70 volume percent sulphuric acid may be used. The solution should be contained as in **A-1.2**. It is operated at about 50°C. To simplify the removal of scale from beryllium copper, all traces of grease and fingerprints should be removed prior to heat treatment.

A-1.3 'Fire Off' or Scaling Dip

The dip is used for removing heavy oxide layers on small parts which may be handled in bulk. Its composition may vary depending on the purpose for which it is used. A typical example is the following:

Sulphuric acid	0 to 30 volume, percent
Nitric acid	15 to 60 volume, percent
Water	Remainder

This solution should be contained in a tub using the same precautions as for bright dips. The tub should be cooled by an outer water jacket and properly ventilated.

NOTE — Lead sulphate may form on the small globules of lead present in leaded brass, which may cause porosity and blistering of subsequent plate. Dilute nitric acid (without sulphuric acid being present) had been successfully used, as has fluoboric acid.

A-1.4 Dichromate Pickle

This pickle is used to remove red colour of stains on the work after the sulphuric acid pickle as described in **A-1.2**. It is not recommended for use prior to plating as it leaves a passive surface. However, this passivity can be removed by a dip in concentrated hydrochloric acid and rinsed in water before proceeding further.

Sulphuric acid	10 to 20 volume, percent
Sodium dichromate ($Na_2Cr_2O_7$, $2H_2O$)	15 to 30 g/l
Water	Remainder

This solution should be isolated from all plating tanks to avoid contamination of solutions. It should be contained, as the sulphuric pickle, in a lead lined tank or in a tub. It should be operated at room temperature. Time required is about 1 minute.

A-1.4.1 The function of the acid is to dissolve the oxide, while the dichromate oxidizes copper from the cuprous to the cupric state. Thus, if a particular brass shows a preferential dissolving of zinc, it is because there is not sufficient dichromate to oxidize the copper at the rate the zinc is being dissolved. If the scale removal is too low, this is because there is not enough acid. Likewise, as the solution warms up in use, the time of immersion may be reduced. However, as dissolved salts building up in the bath, its activity decreases; but its use may be continued for some time by appropriate additions of acid and dichromate, bearing in mind that the function of the dichromate is to oxidize and thus apped up the removal of copper and that the correct proportions for the particular alloy being treated and the particular results desired may be judged by the appearance of the treated metal. In general, too-red appearance indicates not enough dichromate.

A-1.5 Nitric-Phosphoric Acid Pickle

This pickle is used to remove oxides. The parts should be clean and dry before being placed in the solution:

Nitric acid	1 part by volume
Orthophosphoric acid (75 percent)	3 parts by volume

The solution should be contained in a ceramic container. It should be operated at room temperature. Time required is about 1 minute.

A-1.6 Electropolishing

Electropolishing solutions are used to impart a bright smooth finish to the work. It is accomplished by making the work anodic in a solution such as the following:

Orthophosphoric acid (75 percent)	15 to 67 volume, percent
Sulphuric acid	15 to 60 volume, percent
Chromic acid, flakes	2 to 10 weight, percent
Water	9 to 15 weight, percent

This solution should be isolated from all plating tanks.

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BUREAU OF INDIAN STANDARDS

Headquarters :

Manak Bhavan, 9 Bahadur Shah Zafar Marg, New Delhi 110002
Telephones : 331 01 31, 331 13 75

Telegrams : Manaksanstha
(Common to all Offices)

Regional Offices :		Telephone
Central	: Manak Bhavan, 9 Bahadur Shah Zafar Marg, NEW DELHI 110002	331 01 31 331 13 75
Eastern	: 1/14 C. I. T. Scheme VII M, V. I. P. Road, Maniktola CALCUTTA 700054	37 86 62
Northern	: SCO 445-446, Sector 35-C, CHANDIGARH 160036	2 18 43
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